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Do the mechanical and chemical properties of InvisalignTM appliances change after use? A retrieval analysis

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Abstract: AIM: To investigate the mechanical and chemical alterations of Invisalign appliances after intraoral aging. **MATERIALS AND METHODS:** Samples of Invisalign appliances (Align Technology, San Jose, California, USA) were collected following routine treatment for a mean period of 44 ± 15 days (group INV), whereas unused aligners of the same brand were used as reference (group REF). A small sample from the central incisors region was cut from each appliance and the buccal surface was analysed by attenuated total reflectance-Fourier transform infrared (ATR-FTIR) spectroscopy ($n = 5$). Then the appliances were cut ($n = 25$) and embedded in acrylic resin, ground/polished in a grinding polishing machine, and the prepared surfaces were subjected to Instrumented Indentation Testing under 4.9 N load. Force-indentation depth curves were recorded for each group and the following parameters were calculated according to ISO 14577-1; 2002 specification: indentation modulus (E IT), elastic to total work ratio also known as elastic index (IT), Martens Hardness (HM), and indentation creep (C IT). The mean values of the mechanical properties were statistically analysed by unpaired t-test ($\alpha = 0.05$). **RESULTS:** ATR-FTIR analysis confirmed the urethane based structure of the appliances, without important chemical differences attributed to the aging process. INV group showed significantly lower E IT (REF: 2466 ± 20 , INV: 2216 ± 168 MPa), HM (REF: 119 ± 1 , INV: 110 ± 6 N mm⁻²) and higher IT (REF: 40.0 ± 0.3 , INV: $41.5 \pm 1.2\%$), and C IT (REF: 3.7 ± 0.2 , INV: $4.0 \pm 0.1\%$). The increase in IT indicates that INV is a more brittle than REF, whereas the increase in C IT, a decrease in creep resistance. **CONCLUSION:** Despite the lack of detectable chemical changes, intraoral aging adversely affected the mechanical properties of the Invisalign appliance.

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TITLE: Mechanical and chemical alterations of Invisalign appliances after introral use.

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Abstract

AIM: To investigate the mechanical and chemical alterations of Invisalign appliances after intraoral aging.

MATERIALS AND METHODS: Samples of Invisalign appliances (Align Technology, USA) were collected from a selected patient after intraoral placement for a mean period of 44 ± 15 days (group INV), whereas unused aligners of the same brand were used as reference (group REF). A small sample from the central incisors region was cut from each appliance and the buccal surface was analyzed by ATR-FTIR spectroscopy. Then the appliances were cut and embedded in acrylic resin, ground/polished in a grinding polishing machine and the prepared surfaces were subjected to Instrumented Indentation Testing (IIT) under 4.9 N load and 2 s contact time. A total number of 25 force-indentation depth curves were recorded for each group and the following parameters were calculated according to ISO 14577-1; 2002: Indentation Modulus (E_{IT}), Elastic to total work ratio also known as elastic index (η_{IT}) and Martens Hardness (**HM**). Moreover, the Indentation Creep (C_{IT}) was determined employing a tetragonal loading pulse (4.9N force, 120 s). The mean values of the mechanical properties were statistically analyzed by unpaired t-test ($\alpha=0.05$).

RESULTS: ATR-FTIR analysis confirmed the urethane based structure of the appliances, without important chemical differences attributed to the aging process. INV group showed significantly lower E_{IT} (REF: 2466 ± 20 , INV: 2216 ± 168 MPa), HM (REF: 119 ± 1 , INV: 110 ± 6 N/mm²) and higher η_{IT} (REF: 40.0 ± 0.3 , INV: 41.5 ± 1.2 %) and C_{IT} (REF: 3.7 ± 0.2 INV: 4.0 ± 0.1 %). The increase in η_{IT} indicates that INV is a more brittle than REF, while the increase in C_{IT} a decrease in creep resistance.

CONCLUSION: Despite the lack of detectable chemical changes, intraoral aging deteriorated the mechanical properties of the Invisalign appliance.

Introduction

Contemporary orthodontics has seen an increase in patient demands for esthetic orthodontic appliances, such as ceramic brackets, lingual orthodontics and clear aligner therapy (1,2) Esthetics play a significant role in patient's decisions to receive orthodontic treatment: a recent survey found that 33% of young adults would be unwilling to wear visible braces if needed.(3)Another study found that while traditional metal brackets were esthetically acceptable to only 55% of adults, clear aligners were acceptable to over 90%.(2)Clear aligner preference extends to adolescents as well.(4) This demand will likely continue to increase, despite the limitations with certain types of tooth movements. A systematic review published in 2010(4) including two longitudinal trials(5) and many case reports concluded that there was lack of evidence to support or not the use of these appliances.

Treatment efficacy with clear aligners has been reported to be 41% to 59%.(6,7) Great force variation has been claimed during clear aligner therapy, as an aligner with high initial force may be followed by an aligner with a low force, resulting in tooth movement that is not constant.(8)Additionally, as the order of sequential aligners increase, aligner strains relating to force delivery increase.(9) Orthodontic force produced by a thermoplastic material is strongly correlated with its initial mechanical properties and especially stiffness. Therefore any significant changes among different systems or over time in the mouth may have an impact on what aligner system the practitioner chooses to use.(10) Clements et al.(11)found that material properties may effect treatment outcomes, with a stiffer aligner material for a two week activation time showing the best results in defined measurements of occlusal and alignment improvement. Beyond the initial mechanical properties, intraoral aging during mechanotherapy through biofilm modifications and oral environmental conditions might have an adverse effect on materials properties over the treatment time compromising their force delivery capacity and treatment efficacy.

Previous studies(12,13)found substantial morphological variation relative to the as-received specimens involving abrasion at the cusp tips and localized calcification at stagnation sites. Although a clearer understanding of the material properties and aging process may lead to better sequencing of tooth movement) the aforementioned findings are associated only to surface morphological and compositional modifications. Even though there are concerns

that the effect of intraoral mechanism might affect also bulk properties of these appliances(14) there is no till today any relevant information for the bulk properties of Invisalign which dominate the force delivery capacity of these appliances. Therefore the aim of this study is the mechanical and molecular characterization of retrieved Invisalign structures. The hypothesis tested was that exposing the appliances to the intraoral environment does not adversely affect their molecular or mechanical properties.

Materials and Methods

The institutional ethical board approved the protocol and an inform consent was obtained from patients enrolled in the study. Clinically used Invisalign (Align Technology, San Jose, California, USA) appliances for a mean period of 44 ± 15 days were collected from a patient. Small specimens (5 X 5 mm) were cut from visibly intact areas of the buccal surface of central incisor regions of the intraorally aged specimens (INV). As-received aligners, with no history of intraoral exposure, were used as reference (REF). The changes in the molecular composition of the appliances after intraoral exposure were studied by attenuated total reflectance Fourier transform infrared (ATR-FTIR) spectroscopy. The specimens were placed with the buccal surface against the diamond reflective element of a single-reflection ATR accessory equipped with ZnSe lenses (Golden Gate, Specac, Smyrna, GA, USA) and pressed with a sapphire anvil to obtain firm contact. Spectra were acquired employing an FTIR spectrometer(Spectrum GX, Perkin-Elmer Corp, Bacon, UK)operated under the following conditions:4000-650 cm^{-1} range, 4cm^{-1} resolution and 20 scans condition. The depth of analysis was estimated as to 2 μm at 1000cm^{-1} .All spectra were subjected to ATR and baseline corrections.

Specimens from the appliances (n=25) were then embedded in an acrylic resin (VersoCit-2, Struers Ballerup, Denmark) ground with SiC water coolant papers up to 4000grit and polished and (how?) employing a grinding/polishing machine (Dap-V, Struers) and subjected to instrumented indentation testing (IIT), in order to evaluate the following mechanical properties: The indentation modulus (E_{IT}), the elastic index (η_{IT}) defined as the elastic to total work ratio, the Martens Hardness (**HM**) and the indentation creep (C_{IT}). A universal hardness testing machine (ZHU0.2/Z2.5, Zwick Roell, Ulm, Germany) was used with a Vickers indenter. A total number of 25 force - indentation depth curves were taken for each group under 4.9 N load and 2 s contact period for E_{IT} , η_{IT} , **HM** and 120 s contact period for C_{IT} . All

properties were measured according to the international standard ISO14577-1, 2002(15) as follows:

a) The E_{IT} was calculated from the equation:

$$E_{IT} = \frac{1 - (v_s)^2}{\frac{1}{E_r} - \frac{1 - (v_i)^2}{E_i}}$$

where, v_s (0.43) and v_i (0.07) the Poisson's ratios of sample and indenter respectively, E_i the modulus of the indenter (1140 GPa), and E_r the reduced modulus given by the formula:

$$E_r = \frac{\sqrt{\pi}}{2C \sqrt{A_p}}$$

where, C denotes the compliance of the contact and is determined by the slope of dh/dF at maximum test force and A_p is the projected contact area defined according to ISO 14577-2(15).

b) The η_{IT} is given by the equation:

$$\eta_{IT} = \frac{W_{elast}}{W_{total}} * 100\%$$

where W_{elast} is the area under the unloading curve, W_{plast} the area between the loading and unloading curves and W_{total} the sum of elastic and plastic work determined by the total area below the loading curve.

c) For HM using a Vickers indenter, the following formula applies:

$$HM = \frac{F}{26.43 * h^2}$$

where, F stands for the test force and h is the indentation depth under exerted test force.

d) The indentation creep (C_{IT}) was measured by recording the increase in indentation depth between the start and the end of the constant force period. The C_{IT} was determined applying the equation:

$$C_{IT} = \frac{h_2 - h_1}{h_1} * 100$$

where, h_1 and h_2 are the indentation depths at the time $t_1=8$ s and $t_2 = 128$ s respectively.

The results of E_{IT} , η_{IT} , HM and C_{IT} were statistically analyzed by unpaired t-test at 95% level of significance ($\alpha=0.05$).

Results

Fig. 1 demonstrates representative ATR-FTIR spectra from the groups tested. Both groups revealed characteristic bands OH (3380 cm^{-1}), NH (3313 cm^{-1}), aromatic C-H ($3047, 1605, 1597, 812, 766\text{ cm}^{-1}$), CH ($2928, 2853, 1413, 915\text{ cm}^{-1}$), C=O ($1728, 1308\text{ cm}^{-1}$), amide I (C=O of NCO, 1698 cm^{-1}), amide II (NH and C=O of NCO, 1518 cm^{-1}), C-O (1214 and 1205 cm^{-1}) and C-O-C ($1100-1060\text{ cm}^{-1}$). The similarity in reference and in vivo aged spectra denotes that the aged material did not change in molecular composition.

Fig. 2 presents representative force-indentation depth curves of the groups tested. The curve of the in vivo aged material is shifted towards higher indentation depth, implying lower hardness, whereas the unloading curve of the reference group is steeper than the In vivo aged indicating a higher modulus material.

A representative indentation depth-time curve is presented in Fig. 3. The indentation depth increases under constant load, reaching the maximum value approximately 70 s after load application.

The results of mechanical properties tested are presented in Table 1. The specimens of the in vivo aged group showed significantly lower values for E_{IT} , HM and higher for η_{IT} , C_{IT} in comparison with the reference group.

Discussion

According to the results of the present study no molecular changes were identified in the appliances after intraoral aging. However, the mechanical properties tested showed significant differences in comparison with the reference material. Therefore, the null hypothesis must be partially rejected in regards of the mechanical properties.

The results of FTIR analysis comply with previous findings confirming that Invisalign is made of a polyurethane based material (13). However, contrary to previous studies, where compositional differences were found in the intraorally aged materials associated with the in vivo developed biofilm (13,12), no differences were detected between the reference and in vivo aged groups. The retrieved material examined in the present study was lacking of organized biofilm precipitations, facilitating thus, the resolving power of the surface analysis ATR-FTIR method in discriminating structural material changes from the intraorally adsorbed

species. The relative short period of intraoral exposure and the high patients' level of oral care certainly contributed to the absence of organized integuments from the surface of the retrieved appliances. Selection of the outer buccal appliance surfaces for analysis was preferred over the inner surfaces facing the teeth, since they are directly exposed to the oral environment and more subjected to tensile force trajectories.

The lack of differences among the chemical groups between the two testing conditions (reference/in vivo aged) is in agreement with previous results that confirmed no residual monomers and/or byproducts release in artificial saliva(13). Nevertheless, similar spectra may not imply the same composition in polymers, since the degree of polymerization (i.e. the number of monomers in the polymer chain) may vary.

It is well accepted that retrieval analysis obtains critical information as it tests the material in its intended environment.(14) However testing the mechanical properties of in vivo aged Invisalign structures is impossible with the conventional mechanical tests (i.e. tensile, bending, compression and others) as bulky specimens with predefined dimension are required. This limitation is overwhelmed by IIT, where a simple hardness measurement is used to yield a variety of mechanical properties. This method has been already used to characterize the mechanical properties of thermoplastic orthodontic materials.(10)

Based on the experimental outcome of this study, all the mechanical properties tested deteriorated after intraoral aging. The values of indentation modulus (E_{IT}) were found within the range (1500~3000 MPa) reported for orthodontic thermoplastic aligners.(10) From a mechanical standpoint of view, the decrease of modulus implies attenuation of the force delivery capacity by the appliance during intraoral use. The increased elastic index value (η_{IT}) implies that the aged material has been moved towards a more brittle behavior, while the decrease in Martens hardness (HM) indicates a less wear resistant material. Martens hardness was selected against traditional Vickers hardness in order to eliminate the material rebound effect around the indentation, as documented with traditional hardness measurements, providing thus values independent of the indentation size effect(16). The results of creep measurements (C_{IT}) clearly showed that under constant forces developed by opposite dentition, the deformation of the in vivo aged material increased, weakening thus the orthodontic forces exerted.

The deterioration in the mechanical properties tested as documented in the intraorally exposed Invisalign appliances is typical of the polyurethane softening mechanism. This mechanism has been assigned to the two phase microstructure of thermoplastic polyurethanes which are characterized as randomly segmented copolymers consisting of hard and soft segments(17). The soft segments create amorphous regions, whereas the hard segments, composed of polar molecules forming hydrogen bonds, tend to aggregate into ordered domains. The softening mechanism has been associated with the orientation of hard domains perpendicularly to the applied stress and for high strains, fragmentation into smaller pieces to accommodate further strain(17). The ATR-FTIR analysis, though, failed to probe differences in the H-bonding status of the C=O groups, which were identical in the reference and in vivo aged groups.

The degradation of the mechanical properties can be also related to relaxation of residual stresses developed during the manufacturing procedure or leaching of plasticizers during intraoral exposure. However, the later was not confirmed by ATR-FTIR analysis possibly due to the low concentration of the plasticizer. Of course the aforementioned mechanisms can act synergistically towards the degradation of mechanical properties but the verification of their existence requires further experimental research.

From a clinical standpoint, the results of this study indicate that the exerted orthodontic forces are decayed during treatment, but there is no evidence yet that the extent of mechanical degradation slows down tooth movement. If the decrease in mechanical properties affects treatment efficacy, then it will be worthwhile to model the mechanical degradation over intraoral exposure time. This would provide crucial information for the optimization of the treatment outcome with this aligner material.

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Legends of Figures

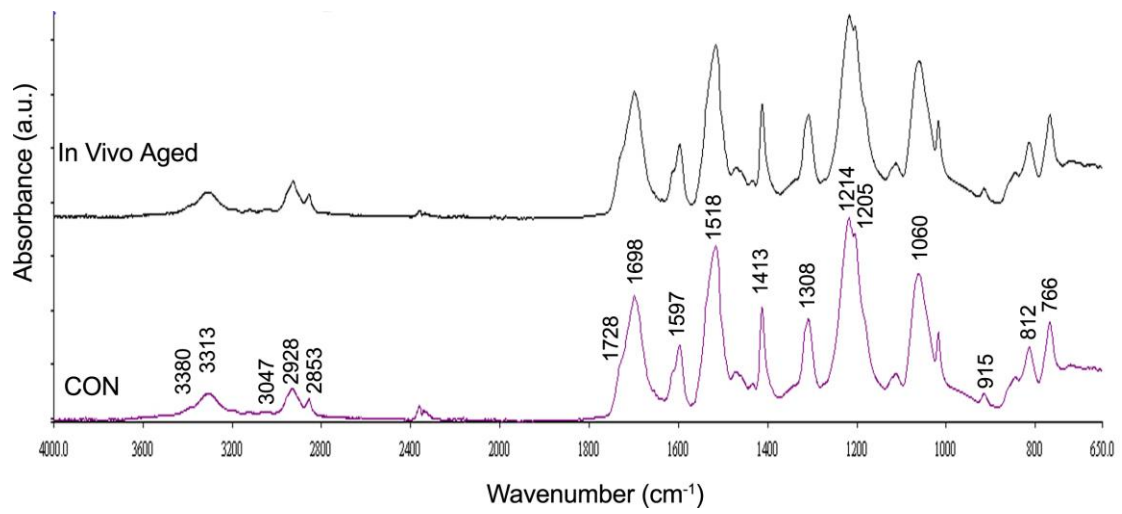


Figure 1. ATR-FTIR spectra of in vivo aged and reference appliances.

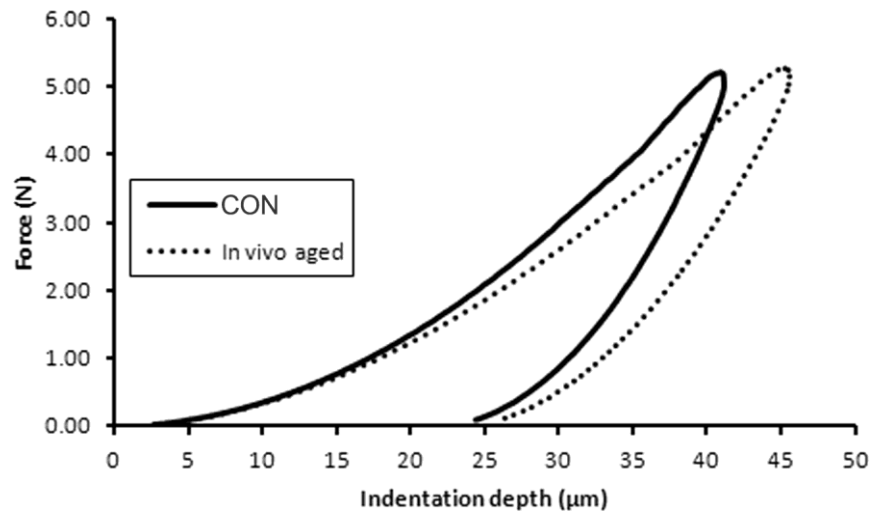


Figure 2. Representative force-indentation depth curves for the reference and in vivo aged groups.

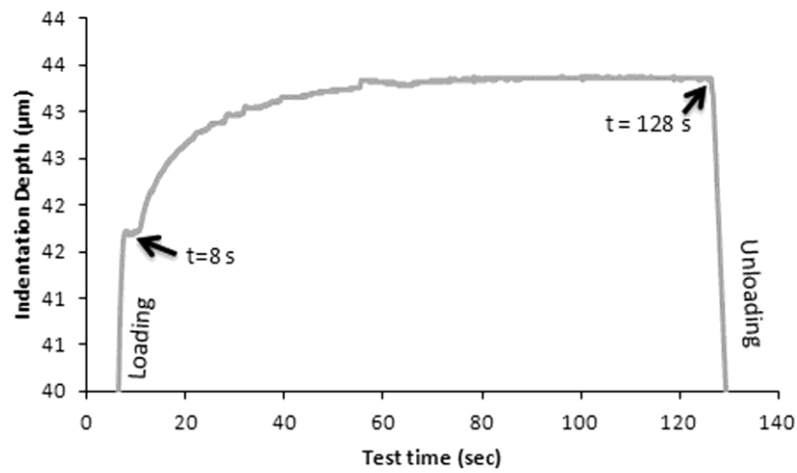


Figure 3. Representative indentation creep curve showing the indentation depth as a function of the test time. The constant load results in increasing indentation depth.

Table 1. Mean values and standard deviations of E_{IT} , η_{IT} HM and C_{IT} for the reference and in vivo aged groups. All properties demonstrated statistical significant differences between the two groups ($p < 0.05$).

Group	E_{IT} (MPa)	η_{IT} (%)	HM (N/mm ²)	C_{IT} (%)
REF	2466±20	40.0±0.3	119±1	3.7±0.2
In Vivo Aged	2216±168	41.51.2	110±6	4.0±0.1
p value	0.008	0.025	0.005	0.028